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REMOVAL PROGRAM QUALITY ASSURANCE
SITE SPECIFIC- SAMPLING AND ANALYSIS PLAN

DOWNER'S GROVE GROUNDWATER INVESTIGATION
DOWNERS GROVE, ILLINOIS

JANUARY 2002

Prepared For:

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Emergency and Enforcement Response Branch
77 West Jackson Boulevard
Chicago, Illinois 60604

TDD No. 0111-010
Document Control No. 195-2F-ABFP

Approvals:

		OM Gazell	1/30/02
Steve Faryan	**Date**	for **Om Patel**	**Date**
On-Scene Coordinator		**START Project Manager**	

GENERAL SITE INFORMATION:

SITE NAME: Downer's Grove Groundwater Investigation	TDD#: 0111-010
SAMPLERS: Ben Maradkel, Geologist, Subcontractors	NO. OF SAMPLES: approx. 60 to 80
LAB: Accutest 495 Technology Center West Building One Marlborough, MA 01752	DATE OF SAMPLING: 2/11/02-2/22/02 DATE SHIPPED: TBD (periodically)

Site Description:

The Site Assessment will focus on the Ellsworth Industrial Park and selected areas east of the industrial park which are located in Downer's Grove, Illinois.

Climatic Limits on Sampling: None

ANALYTICAL INFORMATION:

_____ QA Level I (Field Screening,)

__X__ QA Level II (Laboratory Analysis, Appendix A)

_____ QA Level III (Analyte Specific Laboratory Analysis)

TYPE OF LABORATORY: _____ Commercial __X__ CLP _____ Other

TURNAROUND TIME: (fax) 10 days (committed by Lab. However, Work Plan indicates 14 days.)
(Hardcopy w/data package) 10 days (committed by Lab. However, Work Plan indicates 14 days.)

PURPOSE OF SAMPLING:

(Check all applicable)

__X__ Site Characterization
__X__ Extent of Contamination
_____ Confirm Presence of Suspected Contaminant/Characterize Unknown Contaminant
_____ Disposal/Treatment of Materials
_____ Confirm Efficiency of Existing Treatment Systems
__X__ Other: Assist U.S. EPA in determining the source of the contamination.

MATRIX	NO. OF SAMPLES
Soil	______
Sludge	______
Drum/Tanks/Vats/Containers	______
Wipe	______
Surface Water	______
Groundwater	60 to 80
Air	______
Waste	______
Other: ______	______

TYPE OF SAMPLING:

X Biased (non-random, judgmental) ___ Unbiased (random, systematic grid)

ATTACHMENTS:

1 Sample Location Maps
2 Chain-of-Custody
3 Sampling Form
4 Sample Tracking Log
5 Laboratory SOPs
6 Investigation-Derived Wastes Sampling SOP

SAMPLING METHODS (SOPs):

(Check all applicable)

____	Soil	
____	Sediment	
____	Drum/Tank/Sludge/Waste	
____	Wipe	
____	Surface Water	
X	Groundwater	(Appendix B)
____	Air	
____	Other: ______	

DECONTAMINATION PROCEDURES (SOPs):

X	Personnel	(Appendix C)
____	Equipment	
X	Instrument	(Appendix C)
____	Radiation	

DECONTAMINATION MATERIALS AND INVESTIGATIVE DERIVED WASTE:

All used PPE materials will be properly contained, bagged, and left on site to be disposed of at the discretion of the U.S. EPA.

REFERENCE SITES ON THE INTERNET:

Environmental Response Team (ERT)
www.ert.org
www.ert.org/respns_resres/sops.asp

ATSDR
www.atsdr.cdc.gov/atsdrhome.html

U.S. EPA SW846 Analytical Test Methods
www.epa.gov/epaoswer/hazwaste/test/main.html

U.S. EPA AMTIC Air Toxic Methods
www.epa.gov/ttnamti1/airtox.html

Environmental Test Methods/Guidelines
www.epa.gov/cpahome/standards.html

MSDS
www.msdsonline.com
msms.pdc.cornell.edu/issearch/msdssrch.html
hazard.com/msds/index.html

NIOSH Pocket Guide
www.cdc.gov/niosh/npg/npgdname.html

NIOSH Manual of Analytical Methods
www.cdc.gov/niosh/nmam/nmammenu.html

NAERG
hazmat.dot.gov/gydebook.html

SURFACE WATER / GROUNDWATER / POTABLE WATER SAMPLES

No. of Surface Samples 0 No. of Well Samples 0
No. of Shallow Groundwater Samples 60-80 No. of Trip Blanks 1 per sample cooler
Detection Limit 1 ppb
No. of Duplicates & Equipment Blanks 1 per 10 project samples per parameter

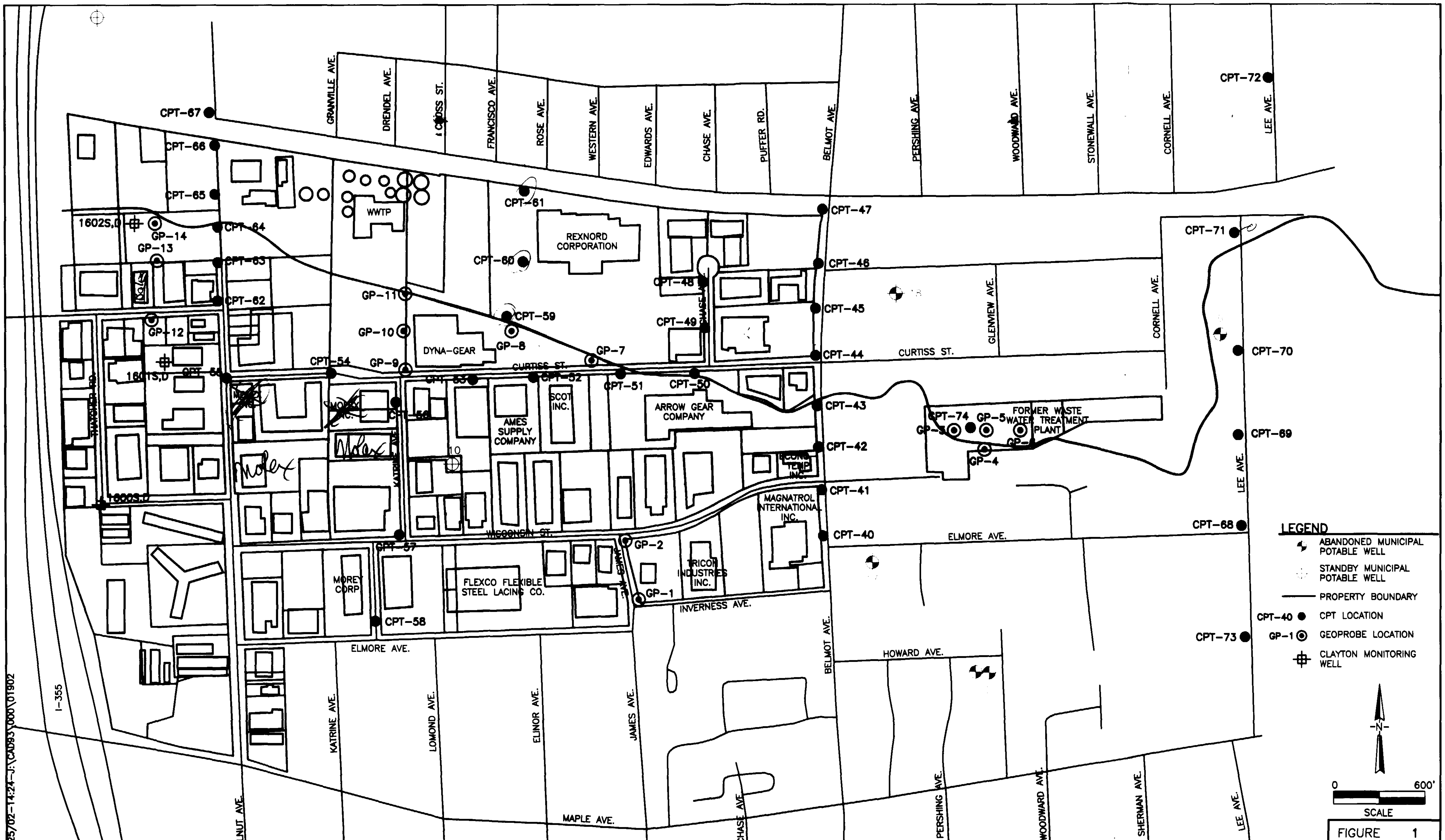
ANALYSIS	NO. OF SAMPLES INCLUDE DUPL. & BLANKS	NO. OF CONTAINERS PER SAMPLE	TOTAL NO. OF CONTAINERS		PRESERVATIVES REQUIRED	LABORATORY METHOD
Semivolatile Organics	0	x 2		80 oz. amber	Ice	
PCB/Pesticides	0	x 2		80 oz. amber	Ice	
Volatile Organics	60 to 80	x 2	120 to 160	40 ml. glass	Ice, 1 ml HCl	SW-846-8260
Dioxin	0	x 2		80 oz. amber	Ice	
Metals	0	x 1		1 liter HDPE	Ice, 5 ml HNO_3	
Cyanide	0	x 1		1 liter HDPE	Ice, 5 ml HNO_3	
Other	0	x ____		____________	___________	

TOTAL NUMBER OF CONTAINERS REQUIRED

__________ 80 oz. amber
180 40 ml. glass
__________ 1 liter HDPE
__________ 8 oz. glass
__________ Other: ____

Attachment 1

Sample Location Map



NOTE:
BASE FIGURE BASED ON "PRIVATE WELL SAMPLING PROGRAM DOWNERS GROVE AND LISLE ILLINOIS" JULY 19, 2001 PREPARED BY PARSONS ENGINEERING SCIENCE, INC.

WESTON
MANAGERS DESIGNERS/CONSULTANTS

750 E. Bunker Ct.
Suite 500
Vernon Hills, Illinois
60061

FIGURE 1

SITE PLAN AND PROPOSED SAMPLING LOCATIONS
USEPA
DOWNERS GROVE SITE
Downers Grove, Illinois

Attachment 2

Chain of Custody

Chain of Custody forms to be provided by Laboratory upon receipt of sample containers.

Attachment 3

Sampling Form

Groundwater Sampling Form

Downer's Grove Groundwater Investigation

WO#: 12634-001-001-0195-00

Location: ______________________ Date: ________

Samplers: ______________________

Comments (weather, temp, etc.): ______________________

Sample ID	Time	Description of area sampled	Sample Appearance

Sample Location Sketch

Attachment 4

Sample Tracking Log

ACCUTEST LABRATORY
495 Technology Center West (Building one)
Marlborough, MA 01752
Phone (508) 481-6200
FAX (000) 000-0000

Downers Grove Groundwater Investigation

Date Samples Sent	Designation of samples	Lab	Analysis	Date Results Due	Date Results Received	Other Info.

ACCUTEST LABRATORY
495 Technology Center West (Building one)
Marlborough, MA 01752
Phone (508) 481-6200
FAX (000) 000-0000

Downers Grove Groundwater Investigation

Attachment 5

Laboratory SOPs

ACCUTEST

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab Manager: Doug Yargeau DRY

QA Manager: Mark Warren

TITLE: DETERMINATION OF VOLATILE ORGANICS USING GC/MS SYSTEM

REFERENCES: SW846 8260B

1.0 SCOPE & APPLICATION

1.1 The following method describes the analytical procedures which are utilized by Accutest to aquire samples for the analysis of volatile organic compounds.

1.2 This analytical method is designed for nearly all types of samples, regardless of water content, including groundwater, aqueous sludges, oily waste, sediments, and soils.

1.3 The purgeable organics can be quantitated by Gas Chromatograph/Mass Spectrometer (GC/MS) following purge and trap utilizing the internal standard technique.

1.4 The Reporting (RL) is based on the lowest calibration standard. RL'S may vary depending on matrix difficulties and sample volumes or weight and percent moisture. Additionally, RL's will vary between some compounds.

2.0 SUMMARY

2.1 This method is performed in accordance with EPA methodologies 8260B and 5030B (purge and trap), from SW-846, 3rd edition.

2.2 An inert gas is bubbled through a 5 ml sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and back flushed with the inert gas to desorb the purgeables onto a gas chromatographic (GC) column.

2.3 The volatile compounds are separated by the temperature programmed GC column and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.4 The peaks detected are qualitated by comparison to characteristic ions and retention times specific to the known target list of compounds.

2.5 Once identified the compound is quantitated by internal standard technique with an average response factor generated from a calibration curve containing a minimum of five points. . Additional points may be added to meet client requirements.

2.6 Additional unknown peaks with a response > 10 % of the closest internal standard may be processed through a library search with comparison to a data base of approximately 70,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

3.0 METHOD DETECTION LIMIT

3.1 The Method Detection Limit (MDL) represents the lowest reportable concentration of an individual analyte that meets the method qualitative identification criteria.

3.2 Method Detection limits (MDLs) are experimentally determined using the procedures described in 40 CFR, Part 136, Appendix B. Actual reported MDLs incorporate the sample volume analyzed and sample dilutions if needed, which may cause MDL variations from sample to sample.

3.3 In general, MDLs are determined through the analysis of at least 7 replicate blank spikes (using the same procedures for sample analysis). The MDL is calculated by multiplying the standard deviation of the replicate concentrations by the appropriate Student's t value (3.143 for 7 replicates). MDLs are determined initially (prior to analysis) and on an annual basis. Refer to the most recent study for current MDLs.

4.0 DEFINITIONS

4.1 ALIQUOT - a measured portion of a sample, or solution, taken for sample preparation and/or analysis.

4.2 BATCH – a group of samples prepared at the same time in the same location using the same method.

4.3 CALIBRATION – the establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards must be prepared using the same type of acid or concentration of acids as used in the sample preparation.

4.4 CALIBRATION STANDARDS – a series of known solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve).

4.5 CONTINUING CALIBRATION – analytical standard run every 12 hours to verify the initial calibration of the analytical system.

4.6 DRY WEIGHT – the weight of a sample based on percent solids. The weight after drying in an oven.

4.7 CONTAMINATION - a component of a sample or an extract that is not representative of the environmental source of the sample. Contamination may stem from other samples, sampling equipment, while in transit, from laboratory reagents, laboratory environment, or analytical instruments.

4.8 FIELD SAMPLE - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

4.9 FIELD BLANK – this is any sample that is submitted from the field and is identified as a blank. This includes trip blanks, rinsates, equipment blanks, etc.

4.10 HOLDING TIME – the elapsed time expressed in days from the date of sampling until the date of its analysis.

4.11 INTERFERENTS – substances which affect the analysis for the analyte of interest.

4.12 GAS CHROMATOGRAPH (GC) - the instrument used to separate analytes on a stationary phase within a chromatographic column. The analytes are volatized directly from the sample (VOA water and low-soil) volatized from the sample extract (VOA medium soil), or injected as extracts (SVOA and PEST). In VOA and SVOA analysis, the compounds are detected by a Mass Spectrometer (MS). In PEST analysis, the compounds are detected by an Electron Capture (EC) detector. In the screening procedure (all fractions), the Flame Ionization Detector (FID) is used as the detector.

4.13 INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer or electron capture detector to the target compounds.

4.14 INTEGRATION TIME RANGE - the retention time at the beginning of the area of integration to the retention time at the end of the area of integration.

4.15 INSUFFICIENT QUANTITY - when there is not enough volume (water sample) or weight (soil/sediment) to perform any of the required operations: sample analysis or extraction, percent moisture, MS/MSD, etc.

4.16 MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is not synonymous with phase (liquid or solid).

4.17 MATRIX EFFECT - in general, the effect of a particular matrix (water or soil/sediment) on the constituents with which it contacts. This is particularly pronounced for clay particles which may adsorb chemicals and catalyze reactions. Matrix effects may prevent extraction of target analytes, and may affect surrogate recoveries. In addition, non-target analytes may be extracted from the matrix causing interferences.

4.18 MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

4.19 MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

4.20 METHOD BLANK - an analytical control consisting of all reagents, internal standards, and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

4.21 PERCENT DIFFERENCE (%D) - As used in this SOW and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

4.22 PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105 °C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

4.23 PURGE AND TRAP (DEVICE) - analytical technique (device) used to isolate volatile (purgeable) organics by stripping the compounds from water or soil by a stream of inert gas, trapping the compounds on an adsorbent such as a porous polymer trap, and thermally desorbing the trapped compounds onto the gas chromatographic column.

4.24 PURGEABLES - volatile compounds.

4.25 REAGENT WATER - water in which an interferant is not observed at or above the minimum quantitation limit of the parameters of interest. Accutest uses deionized water (municipal water which passes through Accutest's DI treatment system).

4.26 RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. In contrast, see percent difference.

4.27 RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation:

$$RRF = \frac{A_x}{A_{is}} x \frac{C_{is}}{C_x}$$

Where,

A = area of the characteristic ion measured
C = concentration, or amount (mass)
is = internal standard
x = analyte of interest

4.28 RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

$$RRT = \frac{RT_c}{RT_{is}}$$

Where,

RT_c = Retention time for the volatile target or surrogate compound in continuing calibration.
RT_{is}= Retention time for the internal standard in calibration standard or in a sample.

4.29 RESPONSE - or Instrumental Response: a measurement of the output of the GC detector (MS, EC, or FID) in which the intensity of the signal is proportionate to the amount (or concentration) detected. Measured by peak area or peak height.

4.30 SOIL - used herein synonymously with soil/sediment and sediment.

4.31 SURROGATES (Surrogate Standard) – for volatiles, semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

4.32 TWELVE-HOUR TIME PERIOD - The twelve (12) hour time period for GC/MS system instrument performance check, standards calibration (initial or continuing calibration), and method blank analysis begins at the moment of injection of the DFTPP or BFB analysis that the laboratory submits as documentation of instrument performance. The time period ends after 12 hours have elapsed according to the system clock. For pesticide/Aroclor analyses performed by GC/EC, the twelve hour time period in the analytical sequence begins at the moment of injection of the instrument blank that precedes sample analyses, and ends after twelve hours have elapsed according to the system clock.

4.33 VOLATILE COMPOUNDS - compounds amenable to analysis by the purge and trap technique. Used synonymously with purgeable compounds.

4.34 RETENTION TIME (RT) - the time a target analyte is retained on a GC column before elution. The identification of a target analyte is dependent on a target compound's retention time falling within the specified retention time window established for that compound. Retention time is dependent on the nature of the column's stationary phase, column diameter, temperature, flow rate, and other parameters.

4.35 DEIONOZED WATER (DI water) - water that has passed through Accutest's deionization system. Used as reagent water (water that an interferant is not observed at or above the minimum quantitation limit of the parameters of interest).

4.36 SPIKE BLANK OR LABORATORY CONTROL SAMPLE (LCS) – A blank spiked with a known concentration of analyte (from a second source from the calibration standard) or an external quality control standard with a known concentration of analyte used to determine accuracy of the method.

5.0 HEALTH & SAFETY

5.1 All safety practices must be followed as outlined in the Accutest Laboratories Chemical Hygiene Plan. Safety glasses, gloves, and lab coats must be worn. All samples, solutions, and extracts must be treated as unknown and potentially hazardous.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIMES

6.1 Collection and Preservation

6.1.1 Soil/Sediment: Refer to SOP MSM207 (Collection and Preservation of Solid Samples for Volatile Organics Analysis by SW846 5035 Methodology).

6.1.2 Aqueous: Samples are collected in certified pre-cleaned 40 ml VOA vials equipped with a teflon-lined silicone septum cap. Samples must be preserved with 1: 1 HCL to a pH of < 2.

Note: The pH of aqueous volatile samples must be checked AFTER analysis. If sample is not properly preserved, this information must be communicated to the client.

6.1.3 Volatile samples must be protected from light and stored segregated from samples for other analyses at a temperature of 4°C ± 2°C from the time of receipt to analysis.

6.2 Holding Time: Samples must be analyzed within 14 days of sampling.

7.0 APPARATUS & MATERIALS

7.1 SYRINGE

7.1.1 10, 25, 50, 100, 500 and 5000 ul graduated syringes, manually held (Hamilton or equiv.).

7.1.2 5 ml glass gas tight syringes with Luerlok end, if applicable to the purging device.

7.2 BALANCE

7.2.1 Analytical balance capable of weighing 0.0001 gram.

7.2.2 Top-loading balance capable of weighing 0.1 g.

7.3 PURGE AND TRAP DEVICES

7.3.1 Tekmar LSC2000 , ALS2016 and O.I. Analytical 4552, 4560, and 4551-a are used for purging, trapping and desorbing the sample into GC column.

7.3.2 The sample purge vessel must be designed to accept 5 ml samples with a water column at least 3 cm deep.

7.3.3 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 inch. The trap must be packed to contain the following absorbents (3-ring):

7.3.3.1 2,6-Diphenylene oxide polymer.
7.3.3.2 Silica gel.
7.3.3.3 Charcoal packing.
7.3.3.4 Or equivalent

7.3.4 The trap should be conditioned according to manufacturer specifications by back flushing with a Helium gas flow of at least 20 ml/min prior to use.

7.3.5 The desorber should be capable of rapidly heating the trap to 180 C fordesorption.

7.3.6 O.I. 4552 is equipped with sample heater for analyzing low-level soils.

7.4 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

7.4.1 Gas Chromatograph.

7.4.1.1 An alytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

7.4.2 Column.

7.4.2.1 For 5890: 75 m x 0.53 mm I.D., 3 um film DB-624; J&W Scientific. Or equivalent. For 6890: 60 m x 0.25 mm I.D., 1.4 um film DB-VRX; J&W Scientific. Or equivalent.

7.4.3 Mass Spectrometer.

7.4.3.1 Capable of scanning from 35-260 amu every second or less utilizing a 70 volt (nominal) electron energy in the electron impact ionization mode.

7.4.3.2 Capable of producing a mass spectrum which meets all the criteria in Table 2 when injecting 50 ng of Bromofluorobenzene(BFB).

7.5 DATA SYSTEM

7.5.1 A computer system is interfaced to the mass spectrometer which allows the continuous acquisition and storage on machine readable media (disc) of all mass spectra obtained throughout the duration of the chromatographic program.

7.5.2 The computer utilizes software which allows searching any GC/MS data file for target analytes which display specific fragmentation patterns.

7.5.3 The Enviroquant data system is capable of quantitation using multipoint calibration and multipoint internal standards.

7.5.4 The recent version of the EPA/NIH mass spectral library (70,000 compounds) is being used for non target peak tentative identification.

7.5.5 Data can be archived to magnetic tape for long term storage.

8.0 STANDARDS & REAGENTS

Note: All applicable standard/reagent preparation information, including vendor, lot number, date of preparation, date of expiration, calculations, and initials must be entered in the appropriate standard/reagent preparation logbook. Vendors typically used by Accutest include Fisher Scientific, VWR, Accustandard, Supelco, Chemservices, Ultra, and ERA. Additional vendors may be utilized as necessary.

8.1 Solvent

8.1.1 Methanol: Purge and trap B&J Brand quality or equivalent. Store apart from other solvents.

8.2 Reagent water

8.2.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

8.2.1.1 Reagent water is generated by either passing tap water through a bed of approximately one pound of activated carbon or by using the water purification system at Accutest which is a series of deionizers and carbon cartridges.

8.3 Stock standard solutions

8.3.1 Commercially prepared standards used:

Accustandard M-502-AR-10X; 2.0 mg/ml in methanol

Accustandard M-502B-10X; 2.0 mg/ml in methanol

Accustandard M8260-Add-10X; 2.0 mg/ml in methanol

Accustandard Acrolein/Acrylonitrile (10000 ug/ml)

Accustandard S-5054 custom mix; 2.0 mg/ml in methanol

Accustandard S-8232 Custom Oxygenate Standard; 2.0 Mg/ml in methanol

Or equivalent standards.

8.3.2 Stock standard solutions (except gases) must be replaced after 6 months or according to manufacturers expiration date if comparison with quality control check samples indicates a problem.

8.3.2.1 The purgeable gases standard should be replaced weekly or sooner if comparison to quality control samples indicates a problem.

8.3.2.2. Stock standard solutions should be stored according to manufacturers specificat Opened ampules should be stored at -10°C except for Acrolein/Acrylonitrile (solution in water) DBW 10/8/01

8.4 Internal Standard and Surrogate Solution.

8.4.1 Four internal standards (see Table 3) are used that exhibit similar analytical behavior to the compounds of interest:

Internal Std: Ultra, Internal Standard Mixture # STM-341N ; 2.0 mg/ml in methanol or equivalent.

Surrogate: Ultra, Surrogate Standard Mixture # STM-330N; 2.0 mg/ml in methanol or equivalent.

8.5 Working standards

8.5.1 A 50 ug/ml working standard is utilized as the calibration, blank spike, and matrix spike solution for the instruments using the Tekmar LSC-II and 2016 . A 200 ug/ml working standard is used for the O.I. 4552 and 4551a autosamplers. A 1:10 dilution of these standards are also prepared to use in the 3 lowest calibration levels.

8.5.1.1 The blank spike and matrix spike solutions are prepared independently from the calibration standards using a different vendor or lot number.

8.5.2 See Table 10 for preparation of working standard.

8.5.3 Bromofluorobenzene (BFB)

8.5.3.1 The BFB is prepared at 25 ug/ml by measuring 25 ul of Ultra 4-Bromofluorobenzene Solution, (#STS-110N, 2000 ug/ml in methanol) into 1975 uls of methanol.

8.5.4 Working standard solutions should be stored at -10°C.

[illegible] 10/8/01

9.0 INTERFERENCES

9.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

9.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

9.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

9.4 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

10.0 PROCEDURE

10.1 CALIBRATION (See Section 10.6.4 before proceeding)

10.1.1 The calibration range covered by the standards is 1, 2, 5, 50, 100, 200, 400 ug/l. (A) The Analyte Reporting Limit can not be lower than the lowest calibration level utilized.

10.1.2 The linear range covered by this calibration is 110 % of highest concentration standard (up to 440 ug/L).

10.1.3 Add 5 ul of internal standard and surrogate solution (see section 8.4) to each standard with a 10 ul syringe. This results in a concentration of 50 ug/L for each internal and surrogate standard. For the O.I. 4552 and 4551-a autosamplers internal and surrogate standards are added from a reservoir containing both at a concentration of 250 ug/ml. 1 ul is added to the standard, sample or blank prior to analyses.

10.1.4 Each analyte is quantitatively determined by internal standard technique using the closest eluting internal standard and the corresponding area of the major ion. See Table 7.

10.1.5 The Response Factor (RF) is defined in section 10.4.1.

10.1.6 Initial calibration

The following criteria must be met for the initial calibration to be valid.

10.1.6.1 The percent relative standard deviation (% RSD) (see section 10.4.2) of calibration check compound (CCC) (see Table 5) must be less than 30 %. The % RSD should be less than 15% for the rest of the compounds for quantitation versus an average response factor to take place. For compounds with % RSD > 15, linear regression or quadratic curve may be used provided the linear coefficient is greater than or equal to 0.990 . A minimum of five calibration levels must be used for a Linear regression and a minimum of six levels must be used for a Quadratic curve.

(A) The ICAL analyzed for Army Corps samples must have a low standard no lower than 5 ppb. Refer to specific Army Corps project QAPP for additional guidance for the ICAL analysis.

10.1.6.2 The minimum average response factor (RF) of the system performance check compound (SPCC) (see Table 5) is 0.300 (>0.10 for Bromoform and 0.10 for chloromethane and 1,1-Dichloroethane).

10.1.6.3 Evaluation of retention times. The relative retention time of each target analyte in each calibration standard should agree within 0.06 relative retention time units.

10.1.7 Continuing calibration (CBCHK)

10.1.7.1 A continuing calibration check standard at mid-level concentration (100 ug/ml) must be acquired every 12 hrs. *

10.1.7.2 The RF's generated for each parameter must be compared to the average RF in the initial calibration for each analyte to determine the percent difference (% D) (see section 10.4.3).

10.1.7.3 The minimum RF of check standard for SPCC compound is 0.300 >0.10 for Bromoform, 0.10 for chloromethane and 1,1-Dichloromethane

10.1.7.4 The % D for CCC must be less than 20.

10.1.7.5 If both of the above specified criteria are met, the continuing calibration is considered valid.

10.1.7.6 If either of the criteria fail, corrective action must be performed. Standard data is evaluated to determine if an analytical system problem exists. If there is problem which does not require making major changes to the system, then those changes are made and the continuing calibration is re-analyzed. If a major problem exists or major changes need to be performed, then the Supervisor is notified for further instruction.

10.1.7.7 If any of the internal standard areas change by a factor of two (- 50% to + 100%) from the last mid-point initial calibration standard, the analytical system must be inspected for malfunctions and corrections will be made, as appropriate.

10.1.7.8 If the retention time for the Internal Standards change by more than 30 seconds from the most recent mid-point initial calibration standard, the system must be inspected for malfunctions. When corrections are made the sample must be reanalyzed.

10.2 ANALYSIS

10.2.1 Instrument conditions.

10.2.1.1 Recommended instrument conditions are listed in Table 1 Modifications are allowed as long as criteria of calibration are met.

10.2.2 Purge and Trap Conditions.

10.2.2.1 Recommended instrument conditions are listed in Table 1.

10.2.3 Daily GC/MS performance check.

*All continuing and initial calibration standards are prepared in volumetric flasks and transferred to 40 ml vials.

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10.1.6.2 The minimum average response factor (RF) of the system performance check compound (SPCC) (see Table 5) is 0.300 (>0.10 for Bromoform and 0.10 for chloromethane and 1,1-Dichloroethane).

10.1.6.3 Evaluation of retention times. The relative retention time of each target analyte in each calibration standard should agree within 0.06 relative retention time units.

10.1.7 Continuing calibration (CBCHK)

10.1.7.1 A continuing calibration check standard at mid-level concentration (100 ug/ml) must be acquired every 12 hrs. *

10.1.7.2 The RF's generated for each parameter must be compared to the average RF in the Initial calibration for each analyte to determine the percent difference (% D) (see section 10.4.3).

10.1.7.3 The minimum RF of check standard for SPCC compound is 0.300 >0.10 for Bromoform, 0.10 for chloromethane and 1,1-Dichloromethane

10.1.7.4 The % D for CCC must be less than 20.

10.1.7.5 If both of the above specified criteria are met, the continuing calibration is considered valid.

10.1.7.6 If either of the criteria fail, corrective action must be performed. Standard data is evaluated to determine if an analytical system problem exists. If there is problem which does not require making major changes to the system, then those changes are made and the continuing calibration is re-analyzed. If a major problem exists or major changes need to be performed, then the Supervisor is notified for further instruction.

10.1.7.7 If any of the internal standard areas change by a factor of two (- 50% to + 100%) from the last mid-point initial calibration standard, the analytical system must be inspected for malfunctions and corrections will be made, as appropriate.

10.1.7.8 If the retention time for the Internal Standards change by more than 30 seconds from the most recent mid-point initial calibration standard, the system must be inspected for malfunctions. When corrections are made the sample must be reanalyzed.

10.2 ANALYSIS

10.2.1 Instrument conditions.

10.2.1.1 Recommended instrument conditions are listed in Table 1 Modifications are allowed as long as criteria of calibration are met.

10.2.2 Purge and Trap Conditions.

10.2.2.1 Recommended instrument conditions are listed in Table 1.

10.2.3 Daily GC/MS performance check.

*All continuing and Initial calibration standards are prepared in volumetric flasks and transferred to 40 ml vials.

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10.2.3.1 Every 12 hours, inject 2 ul (50 ng) of BFB solution directly on column.

10.2.3.2 The GC/MS system must be checked to verify acceptable performance criteria are achieved (see Table 2)

10.2.3.3 This performance test must be passed before any samples, blanks or standards are analyzed.

10.2.3.4 If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are met.

10.2.3.5 The injection time of the acceptable tune analysis, is considered the start of the 12 hour clock.

10.2.4 Daily calibration check

10.2.4.1 See section 10.1.7

10.2.5 Method blank (reagent water)

10.2.5.1 An acceptable method blank must be analyzed for every 12 hour time period.

10.2.5.2 Load 5 ml D.I. water with the 5 ml Luerlok syringe and add 5 ul of 50 ug/ml of internal standard and surrogate mixture to the syringe as a method blank. For O.I. 4551 and 4552 autosamplers, fill a clean 40 ml voa vial with deionized water. Replace teflon lined cap being sure not to leave any air bubbles in vial. Analyze as per 10.2.

10.2.5.3 No compound can be present above the RL (Reporting Limits). The exception to this is Methylene Chloride, 2-Butanone, and Acetone which must be less than 5 x the RL. See Table 8 for RLs.

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10.2.5.4 Surrogates must meet ~~Table 4 criteria~~ or in-house acceptance limits.

10.2.5.5 If the method blank does not meet internal standard, surrogate recovery or contamination criteria, the method blank must be re-analyzed and evaluated before sample analysis can begin. If target compounds are detected in the method blank above the reporting limit, and sample results are non-detects, these results may be reported. 11-5-01

10.2.6 Sample analysis

10.2.6.1 Rinse 5 ml syringes at least three times with organic-free water (reagent water).

10.2.6.2 Establish dilution of sample in order to fall within calibration range (Refer to the Dilution SOP for guidelines in determining need for dilutions and preparation of dilutions).

10.2.6.2.1 from acquired sample data.
10.2.6.2.2 from history program.
10.2.6.2.3 sample characteristics (appearance, sheen, etc.)

10.2.6.3 Water sample

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10.2.6.3.1 pour the sample into the syringe until just overflowing.
10.2.6.3.2 replace the syringe plunger and adjust the sample volume to 5 ml.
10.2.6.3.3 care must be taken to prevent air into the syringe.
10.2.6.3.4 For O.I. 4552 and 4551a autosamplers, place entire 40 ml vial into appropriate position on autosampler.
10.2.6.3.5 Record pH using 0-14 pH paper and record in logbook. (For OI autosamplers, the pH is checked after analysis is complete). (For Tekmar autosamplers, the pH is checked after sample is loaded into purge tube). 10.26.01

10.2.6.4 Sediment/ soil sample

<u>Low-level soil method (Requires separate initial and continuing calibrations)</u> *

10.2.6.4.1 Low level soils are collected in the field preserved in sodium bisulfate. If sample is not preserved – the analyst adds 5 mls of DI water to 5 g of sample (and a stir bar) in a 40 ml voa vial. Soil samples are generally purged on O.I. 4552 instrumentation. Surrogates and IS are loaded onto the instrument (in vials) and automatically injected into the sample. The samples are heated, stirred, and purged onto the trap.
10.2.6.4.2 Alternately, accurately weigh approximately 5 g (or less) sample into clean 40 ml voa vial.
10.2.6.4.3 Add 5 ml reagent water and 1 stir bar to voa vial.
10.2.6.4.4 Place vial into O.I. 4552 autosampler.

<u>Medium-level soil method</u> (Requires separate initial and continuing calibrations) ** DAT 12/12/01

The sample should be extracted in methanol if sample contains analytes above working calibration range or exhibits severe matrix interference.

10.2.6.4.5 weigh 10 g sample into VOA vial containing 10 ml methanol and seal with Teflon lined septum.
10.2.6.4.6 Add 12.5 uls Ultra Surrogate Standard Mixture (2000 ug/ml).
10.2.6.4.7 mix by hand shaking vigorously for 1 minute.
10.2.6.4.8 let settle.
10.2.6.4.9 aliquot proper amount of extract by using gas tight microsyringe.
10.2.6.4.10 add aliquoted sample (extract) to a syringe containing 5 ml reagent water.

10.2.6.5 For medium-level soil analyses, add 5 ul of 50 ug/ml internal standard (I.S.) to syringe containing sample. The concentration of each I.S. should be 50 ug/l without any dilution factors.

10.2.6.6 Attach the syringe to the valve on the TEKMAR ALS and inject into purge vial.

10.2.6.7 For analysis of low-level soils soils, heat the sample vial to 40°C while purging and stirring the sample for 11 minutes with Helium. Water samples are not heated. Sample dry purge time is 2 minutes for both matrices.

10.2.6.8 Desorb the sample for 4 minutes by rapidly heating the trap to 180°C while backflushing with Helium.

* Low level initial and continuing calibration standards are prepared in sodium bisulfate solution.

** Medium level initial and continuing calibration standards are prepared in water containing methanol equivalent to a 1:50 dilution. DAT 12/12/01

10.2.6.9 Bake the trap for 12 minutes at 225°C or at the manufacturers specifications to remove any residual purgeable compounds.

10.2.6.10 If the quantitation value for any analyte exceeds the working range of the GC/MS system, dilute the sample and re-analyze.

10.3 DATA INTERPRETATION

10.3.1 Qualitative identification.

10.3.1.1 The targeted compounds shall be identified by analyst with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. The criteria required for a positive identification are:

10.3.1.2 The sample component must elute at the same relative retention time (RRT) as the daily standard. Criteria is the RRT of sample component must be within ± 0.06 RRT units of the standard.

10.3.1.3 All ions present in the standard mass spectra at a relative intensity greater than 10% (major abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

10.3.1.4 The relative intensities of these ion must agree within ± 30% between the daily standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80%).

10.3.2 Quantitative analysis.

10.3.2.1 When a target compound has been identified, concentration (see section 10.4) will be based on the integrated area of the quantitation ion, normally the base peak (see Table 7).

10.3.2.2 If the sample produces an interference for the primary ion, use a secondary ion to quantitate (see Table 7). This is characterized by an excessive background signal of the same ion which distorts the peak shape beyond a definitive integration. Also an interference could severely inhibit the response of the internal standard ion.This secondary ion must also be used to generate new calibration response factors.

10.3.3 Library search for tentatively identified compounds.

10.3.3.1 If a library search is requested, the analyst should perform a forward library search of NBS mass spectral library to tentatively identify 15 non-reported compounds.

10.3.3.2 Guidelines for making tentative identification are:

10.3.3.2.1 These compounds should have a response greater than 10% of the nearest internal standard. The response is obtained from the integration for peak area of the Total Ion Chromatogram (TIC).

10.3.3.2.2 The search is to include a spectral printout of the 3 best library matches for a particular substance. The results are to be interpreted by analyst.

10.3.3.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.3.3.2.4 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

10.3.3.2.5 The relative intensities the major ions should agree within ± 20%.

10.3.3.2.6 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

10.3.3.2.7 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible background subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.3.2.8 Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 and is to be tabulated on the library search summary data sheet.

10.3.3.2.9 Quantitation will be performed by using the nearest internal standard.

10.3.3.2.10 Report result as estimated.

10.4 CALCULATION

10.4.1 Response Factor (RF)

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

where: As = Area of the characteristic ion for the compound being measured.
Ais = Area of the characteristic ion for the specific internal standard.
Cs = Concentration of the compound being measured (ug/l).
Cis = Concentration of the specific internal standard (ug/l).

10.4.2 Percent Relative Standard Deviation (% RSD)

$$\%RSD = \frac{SD}{RFav} \times 100$$

where: SD = Standard Deviation
RFav = Average response factor from initial calibration.

10.4.3 Percent Difference (% D)

$$\%D = \frac{|RFav - RFc|}{RFav} \times 100$$

where: RFc = RF from continuing calibration (CBCHK)

10.4.4 Concentration (Conc.)

For water:

$$Conc.\ (ug/l) = \frac{Ac \times Cis \times Vp}{Ais \times RFav \times Vi}$$

For soil/sediment (on a dry weight basis):

$$Conc.\ (ug/kg) = \frac{Ac \times Cis \times Vp}{Ais \times RFav \times Ws \times M}$$

Where: Ac = Area of characteristic ion for compound being measured.
Vp = 5 ml (Total Purge Volume)
Vi = Initial volume of water purged (ml).
Ws = Weight of sample extracted (g).
M = (100 - % moisture in sample) / 100 or % solids / 100

10.4.5 Percent Recovery (% R)

$$\%R = \frac{Concentration\ Found}{Concentration\ Spiked} \times 100$$

10.4.6 Relative Percent Difference (RPD)

$$RPD = \frac{|MSC - MSDC|}{(\frac{1}{2})(MSC + MSDC)} \times 100$$

Where: MSC = Matrix Spike Concentration
MSDC = Matrix Spike Duplicate Concentration

11.0 QUALITY ASSURANCE

11.1 QC Requirements Summary

BFB	Every 12 hrs.
Calibration Check std.	Every 12 hrs.
Method blank	Every 12 hrs. and/or every batch
Matrix Spike	one per 20 samples.*
Matrix Spike Duplicate	one per 20 samples.*
Blank Spike	one per batch.*
Surrogate	every sample and standard.
Internal Standard	every sample and standard.

* The maximum number of samples per analytical batch is twenty.

11.2 Daily GC/MS performance check - refer to section 10.2.3

11.3 Daily calibration check - refer to section 10.1.7

11.4 Method blank (reagent water) - refer to section 10.2.5

11.5 Matrix Spike(MS)/Matrix Spike Duplicate(MSD).

11.5.1 One sample is selected at random from each analytical batch of similar matrix types and spiked in duplicate with select compounds to check precision and reproducibility.

11.5.2 Matrix spikes are prepared by spiking an actual sample at a concentration of 50 ug/l or 50 ug/kg based on 5 g dry weight. This is analyzed as outlined in 10.2.

11.5.3 Percent recoveries (% R) (see section 10.4.5) are compared to ~~the acceptance criteria listed in Table 6, or~~ against in-house control limits. 11.5 ...
11.5 ...

11.5.4 A relative percent deviation (RPD) (see section 10.4.6) is calculated and compared to RPD levels presented in Table 6, or against in-house limits (when established).

11.5.5 If matrix spikes do not meet criteria and the QC check sample (blank spike) passed acceptance criteria (see Table 9 or in-house limits), a matrix interference is to be assumed and the data is reportable and must be footnoted.

11.6 Blank Spike.

11.6.1 Reagent water is used for the Blank Spike.

11.6.2 Blank Spikes are prepared by adding 5uls of blank Spike solution (prepared independently and from a second source as the calibration standards) to 5mls reagent water. For O.I. 4552 and 4551a , 10 uls are added to 40 mls. reagent water. See Table 10 for standard preparation.

11.6.3 Percent recoveries are compared to criteria listed in table 6 or against in-house acceptance limits (when established).

11.6.4 Blank spike values are used to verify results when Matrix spike/matrix spike duplicate results indicate a potential problem due to sample matrix.

11.6.5 If blank spike recoveries are biased high, and sample results are non-detected, these results may be reported. If blank spike recoveries are biased low, the samples (and blank spike) ~~must~~ must be re-analyzed. 11-5-01

11.7 Surrogate

11.7.1 All blanks, samples, and standards contain surrogate compounds which are used to monitor method performance.

11.7.2 If the recovery of any surrogate compound does not meet the control limits specified ~~in Table 4, or~~ in-house acceptance limits (when established) the calculation must be checked.

11.7.3 The sample must be reanalyzed if the recovery of any one surrogate is out of control limit.

11.7.4 Above conditions (section 11.7.3) are not required for samples having severe matrix interference problems.

11.7.5 If surrogate recoveries are acceptable upon reanalysis, the data from the reanalysis is reported. If the reanalysis date did not meet the hold time, then both sets of data have to submitted with the reanalysis reported.

11.7.6 If surrogates are still outside control limits upon reanalysis, then both sets of data should be submitted with the first analysis reported.

11.8 Internal Standard.

11.8.1 Retention time for all internal standard must be within ± 30 seconds of the corresponding internal standard in the latest continuing calibration or 100 ug/l standard of initial calibration if samples are analyzed directly following an initial calibration.

11.8.2 The area (Extracted Ion Current Profile) of the internal standard in all analyses must be within 50 to 200% of the corresponding area in the latest calibration standard (12 hr. time period).

11.8.3 If area of internal standard does not meet control limits, the calculations must be checked. If a problem is not discovered, the sample must be reanalyzed.

11.8.4 If areas are acceptable upon reanalysis, the reanalysis data is reported.

11.8.5 If areas are unacceptable upon reanalysis, then both set of data are submitted with the original analysis reported.

11.9 A Precision and accuracy (P&A) study is performed as an initial determination of capability, on an annual basis (continued demonstration of capability – a successful PT result may be used in place of a P&A for continued DOC), and if any significant changes have been made to the instrument. In general, 4 replicates or blank spikes are analyzed using the same procedures and conditions for sample analysis. The percent recovery are compared to ~~Table 9 until~~ in-house control limits are established. If percent recovery criteria are not met, corrective action must be taken to bring the system back into control.

<< Interrupted Transmission >>

14.2 Data may be submitted to the client in a specified electronic format (EDD)

12.0 DOCUMENTATION

12.1 The analytical logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

12.2 If samples require reanalysis, a brief explanation of the reason should be documented in this log.

12.3 The standard preparation logbook must be completed for all standard preparations. All information requested must be completed; the page must be signed and dated by the respective person.

12.4 The Accutest lot number must be cross-referenced on the standard vial.

12.5 The instrument Maintenance logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

12.6 All laboratory logbooks must be reviewed and initialed or signed by the lab manager.

12.7 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

13.0 DATA REVIEW

13.1 The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter of non-conformance.

13.2 A secondary review is performed by department managers, and it includes review of the data produced by their department. All manual calculations, QC criteria, and a comparison of the data package to client specified requirements are checked. The department manager may reject data, initiate reanalysis, take additional corrective action, or reprocess data.

13.3 The laboratory director performs a full tertiary review of the data package following its assembly. This review includes an evaluation of QC data against acceptance criteria and a check of the data package contents to assure that all analytical requirements and specifications were executed.

13.4 Spot-check reviews are performed by the Quality Assurance Officer focusing on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification.

14.0 DATA REPORTING

14.1 A results page including positive results and/or RLs, units, methodology, surrogate recoveries, analysis dates, and data qualifiers are reported. Additional quality control data including calibration summaries, MS/MSD (or duplicate) percent recoveries and RPDs, blank spike recoveries, and method blank results may be reported upon request of the client. Raw data may be reported to the client on request.

14.2 Data may be submitted to the client in a specified electronic format (EDD).

14.3 Once the data is approved by the laboratory manager, it may be accessed by clients via LabLink™.

15.0 POLLUTION PREVENTION & WAST MANAGEMENT

15.1 Pollution Prevention. Users of this method must perform all procedural steps that controls the creation and/or escape of wastes of hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids, or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the Sample and Waste Disposal SOP. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non-hazardous aqueous wastes
15.2.2 Hazardous aqueous wastes
15.2.3 Chlorinated organic solvents
15.2.4 Non-chlorinated organic solvents
15.2.5 Hazardous solid wastes
15.2.6 Non-hazardous solid wastes

16.0 REFERENCES

16.1 SW846 5030B

16.2 SW846 5035

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Table 1

RECOMMENDED OPERATING CONDITION

Gas Chromatograph/ Mass Spectrometer	5890/5970MSD	6890/5973
Carrier Gas(linear velocity)	Helium at 30 cm/sec	1.2 ml/min
Mass range	35 - 260 amu	"
Electron Energy	70 volts (nominal)	"
Scan time	not to exceed 7 sec. per scan	"
Injection port temperature	250degC	"
Source temperature	200 – 250degC	"
Transfer line temperature	250 – 300degC	"
Analyzer temperature	220 – 260degC	"
Initial temperature	36degC	42
Time 1	3 minutes	2 minutes
Column temperature rate	8deg/min.	10deg/min to 80degC 14deg/min to 210degC 16deg/min to 240degC
Final temperature	200degC. 3 min. hold	80degC 2.9min hold
Total run time	30 minutes	20 minutes

Purge and Trap Unit

Water Samples

Purge flow	40 mls/ min.
Purge time	11 min.
Dry Purge	2 min
Desorb preheat	175degC
Desorb	4 min. at 180 degC
Bake	8-12 min. at 225degC
Transfer line	100 – 110degC
Valve temperature	approx. transfer line temp.

Purge and Trap Unit

Soil Samples

Purge flow	40 mls/ min.
Purge time	11 min.
Dry Purge	2 min
Desorb preheat	175degC
Desorb	4 min. at 180degC
Bake	8-12 min. at 225degC
Transfer line	100 – 110degC
Valve temperature	approx. transfer line temp.
Preheat	40degC
Preheat time	3 min.

Table 2

BFB KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 - 40 of mass 95
75	30 - 60 of mass 95
95	Base peak, 100% relative abundance
96	5 - 9% of mass 95
173	<2% of mass 174
174	>50% of mass 198
175	5 - 9% of mass 174
176	>95% and <101% of mass 95
177	5 - 9% of mass 176

Table 3

INTERNAL STANDARD

Internal Standard	Prim./Sec. Ions
Pentafluorobenzene	168
Chlorobenzene-d5	117 / 82, 119
1,4-Difluorobenzene	114 / 63, 88
1,4-Dichlorobenzene-d4	152, 115, 150

Table 4

SURROGATES

Compound	(Prim./Sec. ions)	Control Limit (%)	
		Water	Soil
Dibromofluoromethane	(113)	86 - 118	80 - 120
Toluene-d8	(92 / 91,65)	88 - 110	81 - 117
4-Bromofluorobenzene	(95 / 174,176)	86 - 115	74 - 121

Table 5

Criteria for CCC and SPCC

Initial Calibration: Maximum % RSD for CCC is 30%

Continuing Calibration: Maximum % D for CCC is 20%

Minimum acceptable average relative response factor (RRF) is 0.300 for SPCC (>0.10 for Bromoform, chloromethane & 1,1 Dichloroethane).

Calibration check compounds (CCC)

Volatile
Vinyl Chloride
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene

System Performance Check Compounds (SPCC)

Volatile
Chloromethane
1,1-Dichloroethane
Bromoform
1,1,2,2-Tetrachloroethane
Chlorobenzene

Table 6

MATRIX SPIKE RECOVERY and RSD LIMITS*

Matrix Spike Compound	RPD *		% Recovery limit *	
	Water	Soil	Water	Soil
1,1-Dichloroethene	14	22	61 - 145	59 - 172
Trichloroethene	14	24	71 - 120	62 - 137
Benzene	11	21	76 - 127	66 - 142
Toluene	13	21	76 - 125	59 - 139
Chlorobenzene	13	21	75 - 130	60 - 133

*Advisory Limits only

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Table 7
Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Pentafluorobenzene	ions
Acetone	(43/58)
Acrylonitrile	(53/52,51)
Acrolein	(56/55,58)
Bromochloromethane	(128,49,130)
Bromomethane	(94/96,79)
Carbon disulfide	(76/78)
Chloroethane	(64/66,49)
Chloroform	(83/85,47)
Chloromethane	(50/52,49)
Dichlorodifluoromethane	(85/87,50)
1,1-Dichloroethane	(63/65,83)
2,2-Dichloropropane	(77,97)
1,2-Dichloroethane	(surr.)
1,1-Dichloroethene	(96/61,98)
Iodomethane	(142/127,141)
Methylene chloride	(84/49,51)
Vinyl chloride	(62/64,61)
trans-1,2-Dichloroethene	(96/61,98)
Trichlorofluoromethane	(101/103,66)
Tertiary butyl alcohol	(59/41)
Vinyl Acetate	(43,86)
cis-1,2-Dichloroethene	(96,61,98)
Tetrahydrofuran	(42/71/72)

Chlorobenzene-d5	ions
1,1,2-Trichloroethane	(97,99,61)
Chlorobenzene	(112/114,77)
Ethylbenzene	(106/91)
1,3-Dichloropropane	(76,78)
*Ethyl methacrylate	(69/41,99)
2-Hexanone	(43/58,57)
Styrene	(104/78,103)
Tetrachloroethene	(164/129,131)
Toluene-d8	(surr.)
Xylenes	(106/91)
Chlorodibromomethane	(129/208,206)
1,1,1,2-Tetrachloroethane	(131,133,206)
Bromoform	(173/171,175,252)

1,4-Difluorobenzene	ions
Benzene	(78/52,77)
Bromodichloromethane	(83/85,129)
2-Butanone	(72/57,43)
Carbon tetrachloride	(117/119,121)
2-Chloroethyl vinyl ether	(63/65,106)
Dibromomethane	(93/174,95)
1,4-Dichloro-2-butene	(75/53,89)
1,2-Dichloropropane	(63/62,41)
cis-1,3-Dichloropropene	(75/77,390
trans-1,3-Dichloropropene	(75/77,39)
1,1,1-Trichloroethane	(97/99,117)
Trichloroethene	(130/95,97,132)
Vinyl acetate	(43/86)
Methyl tert butyl ether	(73/57)
1,4-Dioxane	(88/58)
Ethyl acetate	(43/45,61)
Bromofluorobenzene	(surr.)
1,1-Dichloropropane	(75,110,77)
1,2-Dichloroethane	(62,64,98)

1,4-Dichlorobenzene-D4	ions
Isopropylbenzene	(105,120)
Bromobenzene	(156,77,158)
1,2,3-Trichloropropane	(75/110,77,61)
n-Propylbenzene	(91,120)
1,1,2,2 Tetrachloroethane	(83/85,131,133)
2-chlorotoluene	(91,126)
4-chlorotoluene	(91,126)
1,3,5-Trimethylbenzene	(105,120)
t-Butlybenzene	(119,91,134)
sec-Butlybenzene	(105,134)
1,3-Dichlorobenzene	(146/148,111)
1,3-Dichlorobenzene	(146/148,111)
1,2-Dichlorobenzene	(146/148,111)
p-Isopropyltoluene	(119,134,91)
n-Butylbenzene	(91,92,134)
1,2-Dibromo-3-chloropropane	(75,155,157)
1,2,4-Trichlorobenzene	(180,182,145)
Naphthalene	(128)
Hexachlorobutadiene	(225,223,227)

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Toluene	(91/92,65)	1,2,3-Trichlorobenzene	(180,182,145)
4-methyl-2-pentanone	(43/58,100)		
1,2-Dibromoethane			

* non routine target compound

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Table 8

REPORTING LIMITS (RL)

COMPOUND	CAS #	RL WATER (ug/l)	RL SOIL (ug/kg)
ACETONE	67-64-1	5.0	5.0
ACROLEIN	107-02-08	25	25
ACRYLONITRILE	107-13-1	25	25
BENZENE	71-43-2	1.0	2.0
BROMOBENZENE	108-86-1	5.0	5.0
BROMOCHLOROMETHANE	74-97-5	5.0	5.0
BROMODICHLOROMETHANE	75-27-4	2.0	2.0
BROMOFORM	75-25-2	2.0	2.0
BROMOMETHANE	74-83-9	2.0	2.0
2-BUTANONE	78-93-3	5.0	5.0
n-BUTLYBENZENE	104-51-8	5.0	5.0
sec-BUTYLBENZENE	135-98-8	5.0	5.0
tert-BUTYLBENZENE	98-06-6	5.0	5.0
CARBON DISULFIDE	75-15-0	5.0	5.0
CARBON TETRACHLORIDE	56-23-5	2.0	2.0
CHLOROBENZENE	108-90-7	2.0	2.0
CHLOROETHANE	75-00-3	5.0	5.0
CHLOROFORM	67-66-3	2.0	2.0
CHLOROMETHANE	74-87-3	5.0	5.0
2-CHLOROETHYL VINYL ETHER	110-75-8	5.0	5.0
2-CHLOROTOLUENE	95-49-8	5.0	5.0
4-CHLOROTOLUENE	106-43-4	5.0	5.0
DIBROMOCHLOROMETHANE	124-48-1	2.0	2.0
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	5.0	5.0
1,2-DIBROMOETHANE	106-93-4	2.0	2.0
DIBROMOMETHANE	74-95-3	5.0	2.0
1,2-DICHLOROBENZENE	95-50-1	2.0	2.0
1,3-DICHLOROBENZENE	540-73-1	2.0	2.0
1,4-DICHLOROBENZENE	106-46-7	2.0	2.0
DICHLORODIFLUOROMETHANE	75-71-8	2.0	2.0
1,1-DICHLOROETHANE	75-34-3	2.0	2.0
1,2-DICHLOROETHANE	107-06-02	2.0	2.0
1,1-DICHLOROETHYLENE	75-35-4	1.0	2.0
cis-1,2-DICHLOROETHYLENE	156-59-2	2.0	2.0
trans-1,2-DICHLOROETHYLENE	156-60-5	2.0	2.0
1,2-DICHLOROPROPANE	78-87-5	2.0	2.0
1,3-DICHLOROPROPANE	142-28-9	5.0	5.0
2,2-DICHLOROPROPANE	594-20-7	5.0	5.0

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Table 8 (cont.)

REPORTING LIMITS (RL)

COMPOUND	CAS #	RL WATER (ug/l)	RL SOIL (ug/kg)
1,1-DICHLOROPROPENE	563-58-6	5.0	5.0
cis-1,3-DICHLOROPROPENE	10061-01-5	0.5	2.0
trans-1,3-DICHLOROPROPENE	10061-02-6	0.5	2.0
ETHYLBENZENE	100-41-4	1.0	2.0
HEXACHLOROBUTADIENE	87-68-3	5.0	5.0
2-HEXANONE	591-78-6	5.0	5.0
IODOMETHANE	74-88-4	5.0	5.0
ISOPROPYLBENZENE	98-82-8	5.0	5.0
p-ISOPROPYLTOLUENE	99-87-6	5.0	5.0
METHYLENE CHLORIDE	75-09-2	2.0	2.0
4-METHYL-2-PENTANONE	108-10-1	5.0	5.0
NAPHTHALENE	99-20-3	5.0	5.0
n-PROPYLBENZENE	103-65-1	5.0	5.0
[illegible]	[illegible]	5.0	5.0
TETRAHYDROFURAN	109-99-9	50.0	50.0
TOLUENE	108-88-3	1.0	2.0
1,2,3-TRICHLOROBENZENE	87-61-6	5.0	5.0
1,2,4-TRICHLOROBENZENE	120-82-1	5.0	5.0
1,1,1-TRICHLOROETHANE	71-55-6	2.0	2.0
1,1,2-TRICHLOROETHANE	79-00-5	2.0	2.0
TRICHLOROETHYLENE	79-01-6	2.0	2.0
TRICHLOROFLUOROMETHANE	75-69-4	2.0	2.0
1,2,3-TRICHLOROPROPANE	96-18-4	5.0	5.0
1,2,4-TRIMETHYLBENZENE	95-63-6	5.0	5.0
1,3,5-TRIMETHYLBENZENE	108-67-8	5.0	5.0
VINYL ACETATE	108-05-4	5.0	5.0
VINLY CHLORIDE	75-01-4	2.0	2.0
m-XYLENE	108-38-3	1.0	2.0
p-XYLENE	106-42-3	1.0	2.0
o-XYLENE	95-47-6	1.0	2.0

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Table 9
SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE-BORE CAPILLARY COLUMN *

COMPOUND	CONC. RANGE (UG/L)	RECOVERY (%)	STANDARD DEVIATION OF REC.	PERCENT RSD
ACETONE	ND **	ND	ND	ND
ACROLEIN	ND	ND	ND	ND
ACRYLONITRILE	ND	ND	ND	ND
BENZENE	0.1-10	97	6.5	5.7
BROMOBENZENE	0.1-10	100	5.5	5.5
BROMOCHLOROMETHANE	0.5-10	90	5.7	6.4
BROMODICHLOROMETHANE	0.1-10	95	5.7	6.1
BROMOFORM	0.5-10	101	6.4	6.3
BROMOMETHANE	0.5-10	95	7.8	8.2
2-BUTANONE	ND	ND	ND	ND
n-BUTLYBENZENE	0.5-10	100	7.6	7.6
sec-BUTYLBENZENE	0.5-10	100	7.6	7.6
tert-BUTYLBENZENE	0.5-10	102	7.4	7.3
CARBON DISULFIDE	ND	ND	ND	ND
CARBON TETRACHLORIDE	0.5-10	84	7.4	8.8
CHLOROBENZENE	0.1-10	98	5.8	5.9
CHLOROETHANE	0.5-10	89	8.0	9.0
CHLOROFORM	0.5-10	90	5.5	6.1
CHLOROMETHANE	0.5-10	93	8.3	8.9
2-CHLOROETHYL VINYL ETHER	ND	ND	ND	ND
2-CHLOROTOLUENE	0.1-10	90	5.6	6.2
4-CHLOROTOLUENE	0.1-10	99	8.2	8.3
DIBROMOCHLOROMETHANE	0.1-10	92	6.5	7.0
1,2-DIBROMO-3-CHLOROPROPANE	0.5-10	83	16.6	19.9
1,2-DIBROMOETHANE	0.5-10	102	4.0	3.9
DIBROMOMETHANE	0.5-10	100	5.6	5.6
1,2-DICHLOROBENZENE	0.1-10	93	5.8	6.2
1,3-DICHLOROBENZENE	0.5-10	99	6.8	6.9
1,4-DICHLOROBENZENE	0.2-20	103	6.6	6.4
DICHLORODIFLUOROMETHANE	0.5-10	90	6.9	7.7
1,1-DICHLOROETHANE	ND	ND	ND	ND
1,2-DICHLOROETHANE	ND	ND	ND	ND
1,1-DICHLOROETHYLENE	ND	ND	ND	ND

Table 9 (cont.)

COMPOUND	CONC. RANGE (UG/L)	RECOVERY (%)	STANDARD DEVIATION OF REC.	PERCENT RSD
trans-1,3-DICHLOROPROPENE	ND	ND	ND	ND
ETHYLBENZENE	0.1-10	99	8.4	8.6
HEXACHLOROBUTADIENE	0.5-10	100	6.8	6.8
2-HEXANONE	ND	ND	ND	ND
IODOMETHANE	ND	ND	ND	ND
ISOPROPYLBENZENE	0.5-10	101	7.7	7.6
p-ISOPROPYLBENZENE	0.1-10	99	6.7	6.7
METHYLENE CHLORIDE	0.1-10	95	5.0	5.3
4-METHYL-2-PENTANONE	ND	ND	ND	ND
NAPHTHALENE	0.1-100	104	8.6	8.2
n-PROPYLBENZENE	0.1-10	100	5.8	5.8
STYRENE	0.1-100	102	7.3	7.2
1,1,1,2 TETRACHLOROETHENE	ND	ND	ND	ND
1,1,2,2-TETRACHLOROETHANE	0.1-10	91	5.7	6.3
TETRACHLOROTOLUENE	ND	ND	ND	ND
TOLUENE	0.5-10	102	8.1	8.0
1,2,3-TRICHLOROBENZENE	0.5-10	109	9.4	8.6
1,2,4-TRICHLOROBENZENE	0.5-10	108	9.0	8.3
1,1,1-TRICHLOROETHANE	0.5-10	98	7.9	8.1
1,1,2-TRICHLOROETHANE	0.5-10	104	7.6	7.3
TRICHLOROETHYLENE	0.5-10	90	6.5	7.3
TRICHLOROFLUOROMETHANE	0.5-10	89	7.2	8.1
1,2,3-TRICHLOROPROPANE	0.5-10	100	15.0	14.4
1,2,4-TRIMETHYLBENZENE	0.5-10	99	8.0	8.1
1,3,5-TRIMETHYLBENZENE	0.5-10	92	6.8	7.4
VINYL ACETATE	ND	ND	ND	ND
VINLY CHLORIDE	0.5-10	98	6.5	6.7
m-XYLENE	0.1-10	97	6.3	6.5
p-XYLENE	0.5-10	104	8.0	7.7
o-XYLENE	0.1-31	103	7.4	7.2

*** Criteria from SW846 method 8260B**
**** ND: not determined**

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Table 10

WORKING STANDARD PREPARATION

Internal standard and Surrogate mixture	LSC-2000 (50ug/ml)	O.I. 4552/4551a (250 ug/ml)
Internal Standard (2000 ug/ml)	50 ul	250 ul
Surrogates (2000 ug/ml)	50 ul	250 ul
Methanol	1900 ul	1500 ul
Final volume	2000 ul	2000 ul

Calibration mixture	LSC-2000 (50 ug/ml)	O.I. 4552/4551a (200 ug/ml)
Accustandard M502-AR-10X (2000ug/ml)	50 ul	200 ul
Accustandard M502-B 10x (2000ug.ml)	50 ul	200 ul
Accustandard S-5054 (2000ug/ml)	50 ul	200 ul
Accustandard M-603-10x (10000ug/ml)	50 ul	200 ul
Accustandard S-8232 (2000ug/ml)	50 ul	200 ul
Methanol	1750 ul	1000 ul
Final volume	2000 ul	2000 ul

Blank Spike/ Matrix Spike (2nd source)	LSC-2000 (50 ug/ml)	O.I. 4552/4551a (200ug/ml)
Restek 502.2 Calibration Mix 1 (2000ug/ml)	25ul	100 ul
Restek 502.2 Cal 2000 Megamix (2000ug/ml)	25ul	100 ul
Methanol	950ul	800 ul
Final volume	1000ul	1000 ul

Attachment 6

Investigation-Derived Wastes Sampling SOP

SUPERFUND TECHNICAL ASSESSMENT RESPONSE TEAM
STANDARD OPERATING PROCEDURE

SOP 406
INVESTIGATION-DERIVED WASTES SAMPLING

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) provides Roy F. Weston, Inc. (WESTON®), Superfund Technical Assessment Response Team (START) field personnel with general investigation-derived waste sampling guidelines and strategies.

Sampling guidelines and strategies may vary depending upon site specific conditions, equipment and procedural limitations.

2.0 MATERIALS REQUIRED

The following materials are required for investigation derived waste sampling:

- Sampling jars/bottles (check with lab for necessary volume)
- Proper personal protective equipment (PPE)
- Disposable sampling scoops
- Proper level of respiratory protection
- Scissors

3.0 SAFETY PRECAUTIONS

Investigation derived wastes represent a significant threat for personal exposure. When sampling investigation-derived wastes, consult PPE requirements outlined in the site health and safety plan (HASP) regarding the waste source. Sampling activities performed in Level B PPE will effectively minimize exposure risks. For further guidance, consult Material Safety Data Sheets (MSDS), Occupational Safety and Health Association (OSHA) regulations, and corporate health and safety procedures.

4.0 SAMPLING PROCEDURES

To accurately characterize the investigative derived waste, collect a representative sample of the waste. Representative sampling maximizes resources and cost-effectiveness. *Note: If previous sampling analyses accurately characterize the waste, investigation-derived waste sampling may be unnecessary.*

4.1 Personal Protective Equipment

Procedure:

1. Dress in proper PPE as per the site HASP.
2. Select the sample container(s) based on waste, needed volume, and lab specifications.
3. Cut approximately 5-inch x 5-inch squares of each representative PPE piece (i.e. Tyvek®, booties, Saranex®, gloves, etc.).
4. Place the representative pieces of PPE in the proper sample container(s).
5. Label the sample container(s) with the appropriate markings.
6. Repeat steps 1-5 for non-disposable sampling equipment (i.e. drum thieves).

4.2 Decontamination Water/Purged Well Water

Procedure:

1. Dress in proper PPE as per the site HASP.
2. Select the sample container(s) based on waste, needed volume, and lab specifications.
3. Check analysis requirements and lab specifications for preservative information.
4. Submerge the sample container(s) and collect the specified volume.
5. Label the sample container(s) with appropriate markings.

5.0 REFERENCE

EPA. 1991. *Compendium of Emergency Response Team (ERT) Chip, Wipe and Sweep Sampling and Drum Sampling Procedures.* Office of Solid Waste and Emergency Response, Washington, DC. EPA/540/P-91/008.

Appendix A

QA Level II

QA LEVEL II
Laboratory Data Package Deliverable Requirements

A. Metals

1. Case Narrative: -Observations and/or Problems.
   -Methods used.

2. Sample Holding Times: -Analysis dates.

3. Analytical Results for:
   - Samples.
   - Standards: -Number of standards.
     -Concentration of standards.
   - Initial Calibration: % Recoveries (%R) of true value.
   - Continuing Calibrations: % Recoveries (%R) of true value.
   - Calibration Blanks.
   - Method Blank(s): -Detection limit.
     -Amount detected in blank.
   - Matrix Spike/Matrix Spike Duplicate, if applicable: -% Recoveries (%R)
     -% Difference (%D)
   - Laboratory Control Samples.
   - ICP Interference Check Samples, if applicable.
   - ICP Serial Dilutions, if applicable.
   - Standard Additions, if applicable.

4. Copies of Raw Data for all Analytical Results: Peak areas for all items in No. 3.

5. Compound Quantitation Calculations: -Dilutions.
   -Concentrations.
   -Dry weights.
   -Etc.

6. Chain-of-Custody (COC): Photocopy of COC form.

B. Pesticides by Gas Chromatograph (GC)

1. Case Narrative: -Observations and/or Problems.

2. Sample Holding Times: -Extraction dates.
   -Analysis dates.

3. Analytical Results for:
   - Samples: Verification of positive hits by GC/MS or dissimilar column.

   - Initial Calibration: -Pesticide standard retention time windows and standard Rts.
     -Response factors (RF).
     -% relative standard deviation (%RSD) of response factors for aldrin, endrin, DBC, DDT, DCB.
     -% Breakdown for endrin and 4,4-DDT in Evaluation Standard Mix B.
     -Surrogate compound retention times.

   - Continuing Calibrations: -Retention times (RT).
     -Response factors (RF).

-%Relative standard deviation (%RSD) of response factors for aldrin. endrin, DBC, DDT, DCB.
-Analytical sequence.

- Method Blank(s): -Detection limit.
  -Amount detected in blank.
  -Calibration blank.

- Surrogate Recoveries: -% recovery.
  -Control limits.
  -List compound(s) used as surrogate.

4. Copies of Raw Data for all Analytical Results:
   -Sample chromatograms and ion spectra for GC.
   -Enhanced or background subtracted chromatograms and ion spectra for GC.
   -Standard chromatograms and ion spectra for GC.

5. Compound Quantitation Calculations: -Dilutions.
   -Concentrations.
   -Dry weights.
   -Etc.

6. Chain-of Custody: Photocopy of COC form.

C. Organics by GC/MS

1. Case Narrative: -Observations and/or Problems.
   -Methods used.

2. Sample Holding Times: -Extraction dates.
   -Analysis dates.

3. Analytical Results for:
   - Samples: -Relative retention times.
     -Mass Spectra.
     -Ion chromatograms.

   - GC/MS Tuning: BFB/DFTPP ion abundances.

   - Initial Calibration: -Retention times (RT).
     -Response factors (RF and RRF).
     -% relative standard deviation (%RSD).

   - Continuing Calibrations: % difference from initial standard.

   - Method Blank(s): -Detection limits.
     -Amount detected in blank.

   - Surrogate Recoveries: -% recoveries.

   - Tentatively Identified Compounds, if applicable: Ion relative intensities.

4. Copies of Raw Data for all Analytical Results:
   -Sample spectra.
   -Enhanced or background subtracted spectra.

-Standard spectra. (For tentative identified compounds, provide a reference mass spectra from the spectral library.)

5. Compound Quantitation Calculations: -Dilutions.
   -Concentrations.
   -Dry weights.
   -Etc.

6. Chain-of Custody: Photocopy of COC form.

D. Polychlorinated Biphenyls (PCBs) by GC.

1. Case Narrative: -Observations and/or Problems.
   -Methods used.

2. Sample Holding Times: -Extraction dates.
   -Analysis dates.

3. Analytical Results for:
   - Samples: -Retention time windows.
   - Initial Calibration: -3-point calibration for each aroclor of interest.
     -Response factors (RF).
     -% relative standard deviation (%RSD).
   - Continuing Calibrations: % difference from initial standard.
   - Method Blank(s): -Detection limits.
     -Amount detected in blank.
   - Matrix Spike/Matrix Spike Duplicate, if applicable: % recovery.
   - Surrogate Recoveries: -% recovery.
     -Control limits.
     -List compound(s) used as surrogate.

4. Copies of Raw Data for all Analytical Results:
   -Sample chromatograms.
   -Enhanced or background subtracted chromatograms.
   -Standard chromatograms.

5. Compound Quantitation Calculations: -Dilutions.
   -Concentrations.
   -Dry weights.
   -Etc.

6. Chain-of Custody: Photocopy of COC form.

E. Miscellaneous Analysis

1. Case Narrative: -Observations and/or Problems.
   -Methods used.

2. Sample Holding Times: -Extraction dates.
-Analysis dates.

3. Analytical Results for:
   - Samples.
   - Calibration or Standardization.
   - Duplicates.
   - Reagent Blank Results.
   - Spike Recoveries.

4. Copies of Raw Data for all Analytical Results.

5. Compound Quantitation Calculations: -Dilutions.
-Concentrations.
-Dry weights.
-Etc.

6. Chain-of-Custody: Photocopy of COC form.

F. QA/QC Analytical Methods Reference

Analytical Parameter	Analytical Method
VOCs	**SW846 1311/8260**
SVOCs	**SW846 1311/8270**
PCBs	**SW846 8082**
Pesticides	**SW846 8081**
Metals (total) **Metals (TCLP)** **Mercury**	**SW846 6010 or 6020** **SW846 1311** **SW846 7470 or 7471**
Asbestos (PLM, TEM)	**SW846 9010**
Flash point	**SW846 1010 (closed cup)**
pH	**SW846 9045c**
Dioxin	**SW846 8290**
TPH **(DRO,GRO, Oil&Grease)**	**SW846 8015**

Appendix B

Sampling SOPs

SUPERFUND TECHNICAL ASSESSMENT RESPONSE TEAM
STANDARD OPERATING PROCEDURES

SOP 812
CPT GROUNDWATER SAMPLING

1.0 SCOPE AND APPLICATION

The objective of this Standard Operating Procedure (SOP) is to provide general reference information on sampling of groundwater using Cone Penetration Test (CPT) equipment. This guideline is primarily concerned with the collection of water samples from the saturated zone of the subsurface. Every effort must be made to ensure that the sample is representative of the particular zone of water being sampled. These procedures are designed to be used in conjunction with analyses for the most common types of groundwater contaminants (e.g., volatile and semi-volatile organic compounds, pesticides, metals, biological parameters).

2.0 METHOD SUMMARY

Groundwater samples may be collected directly through CPT push rods equipped with a sleeved groundwater sampling device approximately 2-feet in length. Groundwater sampling intervals are selected following examination of the stratigraphic log and identification of target water bearing zones. The groundwater sampler is advanced by hydraulically pushing it to the predetermined depth, The groundwater sampler consists of a screen with a retractable outer casing. The screen is opened by pulling back on the CPT rod string thereby exposing the screen to the formation. Sampling can then be performed using several methods. The most common of these are the bailer, peristaltic (suction-lift) pump, non-gas contact bladder pump, and/or inertia pump. Equipment must be decontaminated prior to use and between wells. Sampling may be conducted with any of the above instruments. Care should be taken when choosing the sampling device as some can affect the integrity of the sample depending on the suite of analyses desired. Sampling equipment must also be decontaminated. Sampling should occur in a progression from the least to most contaminated location, if this information is known.

Add Piezometer Method
Decon Procedures

Decon of Cone Water
Collect wash water + analyze

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

The type of analysis for which a sample is being collected determines the type of bottle, preservative, holding time, and filtering requirements. Samples should be collected directly from the sampling device into appropriate laboratory-cleaned containers. Check that a Teflon liner is present in the cap, if required. Attach a sample identification label. Complete a field data sheet, a chain of custody form and record all pertinent data in the site logbook.

Samples shall be appropriately preserved, labeled, logged, and placed in a cooler to be maintained at 4°C. Samples must be shipped well before the holding time is over and ideally should be shipped within 24 hours of sample collection. It is imperative that these samples be shipped or delivered daily to the analytical laboratory in order to maximize the time available for the laboratory to perform the analysis. The bottles should be shipped with adequate packing and cooling to ensure that they arrive intact.

Certain conditions may require special handling techniques. For example, treatment of a sample for volatile organic analysis (VOA) with sodium thiosulfate preservative is required if there is residual chlorine in the water (such as public water supply) that could cause free radical chlorination and change the identity of the original contaminants. However, sodium thiosulfate should not be used ~~if chlorine is not present~~ in the water. Special requirements must be determined prior to conducting fieldwork.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 General

The primary goal of groundwater sampling is to obtain a representative sample of the groundwater body. Analysis can be compromised by field personnel in two primary ways: (1) taking an unrepresentative sample, or (2) by incorrect handling of the sample. There are numerous ways of introducing foreign contaminants into a sample, and these must be avoided by following strict sampling procedures and only utilizing trained field personnel.

4.2 Materials

Samplers and evacuation equipment (bladders, pumps, bailers, etc.) should be limited to those made with stainless steel, Teflon, and glass in areas where concentrations are expected to be at or near the detection limit. The tendency of organics to leach into and out of many materials make the selection of materials critical for trace analyses.

Table 1 discusses the advantages and disadvantages of certain equipment.

5.0 EQUIPMENT/APPARATUS

5.1 General

i. water level indicator
   - electric sounder
   - steel tape

- transducer
- reflection sounder
- airline

a. depth sounder

b. appropriate keys for well cap locks

Table 1: Advantages and Disadvantages of Various Groundwater Sampling Devices

Device	Advantages	Disadvantages
Bailer	The only practical limitations are size and materials. No power source needed. Portable. Inexpensive; it can be dedicated and hung in a well, reducing the chances of cross-contamination. Minimal outgassing of volatile organics while sample is in bailer. Readily available. Removes stagnant water first. Rapid, simple method for removing small volumes of purge water.	Time consuming, especially for large wells. Transfer of sample may cause aeration.
Submersible Pump	Portable; can be used on an unlimited number of wells. Relatively high pumping rate (dependent on depth and size of pump). Generally very reliable; does not require priming.	Potential for effects on analysis of trace organics. Heavy and cumbersome, particularly in deeper wells. Expensive. Power source needed. Susceptible to damage from silt or sediment. Impractical in low yielding or shallow wells. Equipment limited by size and may not be feasible for CPT groundwater sampling.
Non-Gas Contact Bladder Pump	Maintains integrity of sample. Easy to use.	Difficult to clean although dedicated tubing and bladder may be used. Only useful to approximately 100 feet in depth. Supply of gas for operation (bottled gas and/or compressor) is difficult to obtain and is cumbersome.

Suction Pump	Portable, inexpensive, and readily available.	Only useful to approximately 25 feet or less in depth. Vacuum can cause loss of dissolved gases and volatile organics. Pump must be primed and vacuum is often difficult to maintain. May cause pH modification.
Inertia Pump	Portable, inexpensive, and readily available. Rapid method for purging relatively shallow wells.	Only useful to approximately 70 feet or less in depth. May be time consuming to use. Labor intensive.

i. steel brush
ii. Mirco FID or Multi RAE 5 Gas detector (whichever is most appropriate)
iii. logbook
iv. calculator
v. field data sheets
vi. chain-of-custody forms
vii. forms and seals
viii. sample containers
ix. engineer's rule
x. sharp knife (locking blade)
xi. tool box (to include at least: screwdrivers, pliers, hacksaw, hammer, flashlight, adjustable wrench)

i. leather work gloves
ii. appropriate health and safety gear
iii. 5-gallon pail
iv. plastic sheeting
v. shipping containers
vi. packing materials
vii. bolt cutters
viii. Ziploc plastic bags
ix. containers for evacuation of liquids
x. decontamination solutions
xi. tap water
xii. non-phosphate soap
xiii. several brushes
xiv. pails or tubs
xv. aluminum foil
xvi. garden sprayer

xvii. preservatives
xviii. distilled or deionized water

5.2 Bailer

i. clean, decontaminated bailer(s) of appropriate size and construction material
ii. nylon line, enough to dedicate to each well
iii. Teflon-coated bailer wire
iv. sharp knife
v. aluminum foil (to wrap clean bailers)
vi. 5-gallon bucket

5.3 Non-Gas Contact Bladder Pump

i. non-gas contact bladder pump
ii. compressor or nitrogen gas tank
iii. batteries and charger
iv. Teflon tubing – enough to dedicate to each well
v. Swagelock fitting
vi. toolbox supplements – same as submersible pump

5.4 Suction Pump

i. pump
ii. black coil tubing – enough to dedicate to each well
iii. gasoline – if required
iv. toolbox
v. plumbing fittings
vi. flow meter with gate valve

5.5 Inertia Pump

i. pump assembly (WaTerra pump, piston pump); or tubing and check valve assembly
ii. 5-gallon bucket

6.0 REAGENTS

Reagents will be utilized for preservation of samples and for decontamination of sampling equipment. The preservation required is specified by the analysis to be performed. Decontamination solutions are specified in SOP No. 301, Decontamination

Procedures.

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies are needed.

2. Obtain necessary sampling and monitoring equipment.

3. Decontaminate or preclean equipment, and ensure that it is in working order.

4. Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry in accordance with the site-specific health and safety plan.

6. Identify and mark all sampling locations.

7.2 Field Preparation

1. Start at the least-contaminated sample location, if known.

2. Screen headspace of CPT rods with an appropriate monitoring instrument to determine the presence of volatile organic compounds and record in site logbook.

insert TVA 1000
- SOP's to Run
- Hnu to scan at Methane Portable GC.

3. Lower water level measuring device or equivalent (i.e., permanently installed transducers or airline) into well until water surface is encountered.

4. Measure distance from water surface to reference measuring point.

5. Select the appropriate purging and sampling equipment.

7.3 Sampling

Sample withdrawal methods require the use of pumps, compressed air, bailers, and samplers. Ideally, sample withdrawal equipment should be completely inert, economical to use, easily cleaned, sterilized, reusable, able to operate at remote sites in the presence of power resources, and capable of delivering variable rates for sample collection.

There are several factors to take into consideration when choosing a sampling device. Care should be taken when reviewing the advantages or disadvantages of any one device.

7.3.1 Bailer

The positive-displacement volatile sampling bailer (by GPI) is perhaps the most appropriate for collection of water samples for volatile analysis. Other bailer types (messenger, bottom fill, etc.) are less desirable, but may be mandated by cost and site conditions. Generally, bailers can provide an acceptable sample, providing that sampling personnel use extra care in the collection process.

1 Attach a line to the bailer. If a bailer was used for purging, the same bailer and line may be used for sampling.

2 Lower the bailer slowly and gently into the rods taking care not to shake the casing sides or to splash the bailer into the water. Stop lowering at a point adjacent to the screen.

3. Allow bailer to fill and then slowly and gently retrieve the bailer, avoiding contact with the casing, so as not to knock foreign materials into the bailer.

4. Remove the cap from the sample container and place it on the plastic sheet or in a location where it will not become contaminated. See Section 7.7 for special consideration on VOA samples.

5. Begin pouring slowly from the bailer.

6. Filter and preserve samples as required by sampling plan.

7. Cap the sample container tightly and place pre-labeled sample container in a carrier.

8. Log all samples in the site logbook and on field data sheets and label all samples.

9. Package samples and complete necessary paperwork.

10. Transport sample to decontamination zone to prepare it for transport to analytical laboratory.

7.3.2 Non-Gas Contact Bladder Pump

These pumps are suitable for shallow (less than 100 feet) samples. They are somewhat difficult to clean, but may be used with dedicated sample tubing to avoid cleaning. These pumps require a power supply and a compressed gas supply (or compressor). They may be operated at variable flow and pressure rates making them ideal for both purging and sampling, as needed.

Barcelona (1984) and Nielsen (1985) report that the non-gas contact positive displacement pumps cause the least amount of alteration in sample integrity as compared to other sample retrieval methods.

1. Allow CPT rods to recharge after opening sampler.

2. Assemble the appropriate bottles.

3. Turn pump on, increase the cycle time and reduce the pressure to the minimum that will allow the sample to come to the surface.

4. Cap the sample container tightly and place pre-labeled sample container in a carrier.

5. Log all samples in the site logbook and on field data sheets and label all samples.

6. Package samples and complete necessary paperwork.

7. Transport sample to decontamination zone for preparation for transport to analytical laboratory.

8. On completion, remove the tubing from the well and either replace the Teflon tubing and bladder with new dedicated tubing and bladder or rigorously decontaminate the existing materials.

9. Collect non-filtered samples directly from the outlet tubing into the sample bottle.

10. For filtered samples, connect the pump outlet tubing directly to the filter unit. The pump pressure should remain decreased so that the pressure

build-up on the filter does not blow out the pump bladder or displace the filter. For the Geotech barrel filter, no actual connections are necessary so this is not a concern.

7.3.3 Suction Pump

Suction lift, or peristaltic pump, may be used where water levels are within approximately 25 feet of the ground surface. A new length of tubing should be used for each sample attempt.

7.3.4 Inertia Pump

Inertia pumps may be used to collect samples. It is more common, however, to purge with these pumps and sample with a bailer.

1. Allow the CPT rod to recharge after the sampler is opened.

2. Assemble the appropriate bottles.

3. Since these pumps are manually operated, the flow rate may be regulated by the sampler. The sample may be discharged from the pump outlet directly into the appropriate sample container.

4. Cap the sample container tightly and place pre-labeled sample container in a carrier .

5 Log all samples in the site logbook and on field data sheets and label all samples.

6. Package samples and complete necessary paperwork.

7. Transport sample to decontamination zone for preparation for transport to analytical laboratory.

8. Upon completion, remove pump and decontaminate ~~or discard~~, as appropriate.

7.4 Filtering

For samples that require filtering, such as samples which will be analyzed for total metals, the filter must be decontaminated prior to use and between uses. Filters work by two methods. A barrel filter such as the "Geotech" filter works with a bicycle pump, which is used to build up positive pressure in the chamber containing the sample. The sample is then forced through the filter paper (minimum size 0.45 μm) into a jar placed underneath. The barrel itself is filled manually from the bailer or directly via the hose of the sampling pump. The pressure must be maintained up to 30 psi by periodic pumping.

A vacuum type filter involves two chambers, the upper chamber contains the sample and a filter (minimum size 0.45 μm) divides the chambers. Using a hand pump or a Gillian type pump, air is withdrawn from the lower chamber, creating a vacuum and thus causing the sample to move through the filter into the lower chamber where it is drained into a sample jar, repeated pumping may be required to drain all the sample into the lower chamber. If preservation of the sample is necessary, this should be done after filtering.

7.5 Post Operation

After all samples are collected and preserved, the sampling equipment should be decontaminated prior to sampling another location. This will prevent cross-contamination of equipment and monitoring wells between locations.

1. Decontaminate all equipment

2. Replace sampling equipment in storage containers.

3. Prepare and transport water samples to the laboratory. Check sample documentation and make sure samples are properly packed for shipment.

7.6 Special Considerations for VOA Sampling

The proper collection of a sample for volatile organics requires minimal disturbance of the sample to limit volatilization and therefore a loss of volatiles from the sample.

Sample retrieval systems suitable for the valid collection of volatile organic samples are: positive displacement bladder pumps, gear driven submersible pumps, syringe samplers and bailers (Barcelona, 1984; Nielsen, 1985). Field conditions and other constraints will limit the choice of appropriate systems. The focus of concern must be to provide a valid sample for analysis, one which has been subjected to the least amount of turbulence possible.

The following procedures should be followed:

1. Open the vial, set cap in a clean place, and collect the sample during the middle of the cycle. When collecting duplicates, collect both samples at the same time.

2. Fill the vial to just overflowing. Do not rinse the vial, nor excessively overfill it. There should be a convex meniscus on the top of the vial.

3. Check that the cap has not been contaminated (splashed) and carefully cap the vial. Place the cap directly over the top and screw down firmly. Do not overtighten and break the cap.

4. Invert the vial and tap gently. Observe vial for at least 10 seconds. If an air bubble appears, discard the sample and begin again. It is imperative that no entrapped air is in the sample vial.

5. Immediately place the vial in the protective foam sleeve and place into the cooler, oriented so that it is lying on its side, not straight up.

6. The holding time for VOAs is 7 days. Samples should be shipped or delivered to the laboratory daily so as not to exceed the holding time. Ensure that the samples remain at 4°C, but do not allow them to freeze.

8.0 CALCULATIONS

There are no calculations necessary to implement this procedure.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

There are no specific quality assurance activities which apply to the implementation of these procedures. However, the following general QA procedures apply:

- All data must be documented on field data sheets or within site logbooks.

- All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling/operation and they must be documented.

10.0 DATA VALIDATION

This section is not applicable to this SOP.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA and specific health and safety procedures. More specifically, depending upon the site-specific contaminants, various protective programs must be implemented prior to sampling the first well. The site health and safety plan should be reviewed with specific emphasis placed on the protection program planned for the well sampling tasks. Standard safe operating practices should be followed such as minimizing contact with potential contaminants in both the vapor phase and liquid matrix through the use of respirators and disposable clothing.

For volatile organic contaminants:

- Avoid breathing constituents venting from the well.
- Pre-survey the well head-space with an FID/PID prior to sampling.
- If monitoring results indicate organic constituents, sampling activities may be conducted in Level C protection. At a minimum, skin protection will be afforded by disposable protective clothing.

Physical hazards associated with groundwater sampling are:

- Lifting injuries associated with pump and bailer retrieval; moving equipment.
- Use of pocket knives for cutting discharge hose.
- Heat/cold stress as a result of exposure to extreme temperatures (may be heightened by protective clothing).
- Slip, trip, fall conditions as a result of pump discharge.
- Restricted mobility due to the wearing of protective clothing.

12.0 REFERENCE

EPA. 1991. *Compendium of ERT Groundwater Sampling Procedures.* Office of Solid Waste and Emergency Response, Washington, DC. EPA/540/P-91/008.

Attachment: 1

ATTACHMENT 1

WATER LEVEL and WATER QUALITY/WELL PURGING DATA SHEET

Date: ___________

Time: ___________

Water Level and Water Quality / Well Purging Data Sheet

Site Name: ____________________ **Well Number:** ___________ **Sampler:** _______________

Purging Method (Circle One): Poly Bailer Teflon® Bailer Steel Bailer Keck Pump WaTerra® Pump Piston Pump Bladder Pump

Sampling Method (Circle One): Poly Bailer Teflon® Bailer Steel Bailer Bacon Bomb WaTerra® Pump Piston Pump Bottle Submersion

Water Level and Volume Measurements

Measure Point (Circle One): Top of Casing Top of Protective Steel Casing Land Surface (mark measure point)

Depth to Water: ____ft____in **Depth to Bottom:** ____ft____in **Depth to Product:** ____ft____in ____N/A

Height of Water Column (H): ____ft____in (round up for volume calculation)

Gallons per Foot of Depth/Diameter (GPF): 1" **(.041)** 1.5" **(.092)** 2" **(.163)** 2.5" **(.255)** 3" **(.367)** 3.5"**(.500)** 4" **(.653)** 4.5" **(.826)** 5" **(1.020)** 5.5" **(1.234)** 6" **(1.469)** 7" **(2.000)** 8" **(2.611)** 9" **(3.305)** 10" **(4.080)**

One Well Volume: ____**(H)** x ______**(GPF)** = ____Gals **Volume to Purge:** ____Gallons x 3 = ____Gals

Actual Volume Purged: ____Gals (estimated) **Purge Rate:** ____Gals/min (estimated) (volume/time)

Water Quality / Purge Data

Interval	Time	Temp	pH	Conductivity	Turbidity	% Dissolved O_2	PID/FID
Start				mOhms	H M L N	% O_2	units
During				mOhms	H M L N	% O_2	units
During				mOhms	H M L N	% O_2	units
During				mOhms	H M L N	% O_2	units
During				mOhms	H M L N	% O_2	units
During				mOhms	H M L N	% O_2	units
During				mOhms	H M L N	% O_2	units
During				mOhms	H M L N	% O_2	units
Final				mOhms	H M L N	% O_2	units

H - High: Opaque/Muddy/Silty   M - Medium: Translucent/Cloudy   L - Low: Transparent/Cloudy   N - None: Clear/No Visible Silt

Appendix C

Decontamination SOPs

SUPERFUND TECHNICAL ASSESSMENT RESPONSE TEAM
STANDARD OPERATING PROCEDURES

SOP 301
DECONTAMINATION PROCEDURES

1.0 PURPOSE

To provide guidance for the decontamination of equipment used to sample, and install sample, and install sample points (monitor wells, soil borings and test pits), and make field measurements. This operating practice is not intended to be site specific or equipment specific, but to provide guidance in place of non-existent state or federal guidelines.

2.0 DISCUSSION

2.1 Introduction

The objective of decontamination procedures is to provide clean equipment for the retrieval of representative environmental samples. Decontamination procedures differ depending on the nature of the equipment used. The three categories of decontamination procedures are discussed below:

- Intrusive equipment used to install sample points including drilling (tools, augers, rods, etc.) and excavation equipment (backhoes, excavators, etc.).

- Equipment used to measure the characteristics of the media to be sampled including water level, pH, specific conductivity, and temperature probes. This category also includes pumps to purge water.

- Equipment that has contact with the sample to be submitted for laboratory analysis including bailer, split-spoons, hand auger, stainless steel bowls and scoops.

Because items from the first two categories do not contact the sample media that is sent to a laboratory for analysis, the decontamination procedures are less stringent. Dedicated and disposable equipment will be used whenever feasible to limit decontamination and the possibility of cross-contamination. This includes rope, tubing, filterware and, in some cases, soil scoops and bailers.

3.0 PROCEDURES

3.1 Intrusive Equipment

Drilling tools, including augers, rods, drill bits, hand tools, etc. will be steam cleaned prior to use and after each location. Split spoons will also be steam cleaned if not used for sample collection. Backhoe buckets and arms will also be steam cleaned prior to use and between each sample location.

3.2 Field Measurement Equipment

Water level probes will be cleaned using the following procedures:

- Wipe the probe with a paper towel.
- Alconox and potable water wash.
- Deionized water rinse.

Other measurement equipment should be rinsed with deionized water between readings.

Pumps used for well purging shall be decontaminated using the following procedures:

- Alconox and potable water scrub and pump through.
- Potable water rinse and pump through.

Rope and tubing used with the pump will be made of polyethylene and be dedicated (and disposable) to one sample location.

3.3 Sampling Equipment

Equipment used for sample collection include but are not limited to:

- Teflon bailers
- Stainless steel scoops and bowls
- Hand augers
- Split spoons

This equipment will be cleaned using the following procedures:

- Alconox and potable water scrub.

- Thorough potable water rinse.
- Deionized water rinse.
- 10% nitric acid rinse* (1% solution if used on low carbon steel split spoons).
- Deionized water rinse*.
- Acetone (pesticide grade) rinse**.
- Total air dry**.
- Deionized water rinse**.

  * Only if sample is to be analyzed for metals.

  ** Only if sample is to be analyzed for organics.

Sampling instruments should be wrapped in aluminum foil after decontamination to keep clean before sampling.

4.0 DOCUMENTATION

Decontamination efforts should be documented in the field logbook. Decontamination fluids should be disposed of properly. Depending on site conditions, it may be appropriate to contain spent decontamination fluids. In that case, the appropriate vessel (i.e., drum) should be used depending on the ultimate disposition of the material.

5.0 INTERPRETATION

If there are questions on the interpretation or applicability of items in this operating practice, the Project Manager or Technical Manager should be consulted. In the absence of either of those, contact a Section Manager.

6.0 REFERENCES

New Jersey Department of Environmental Protection and energy Field Sampling Procedures Manual, May 1992.

"Standard Practice for Decontamination of Field Equipment Used at Non-radioactive Waste Sites", ASTM Designation D5088-90.